TEXTILE RESEARCH

Publication of

United States Institute for Textile Research, Inc.

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Publication Office: Prince and Lemon Sts., Lancaster, Pa.

Editorial and General Offices: 65 Franklin St., Boston, Mass.

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Vol. IV **November, 1933** **No. 1**

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Entered as second-class matter, November 17, 1932, at the Post Office at Lancaster, Pa., under the Act of Congress of March 3, 1879.

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Issued monthly at Prince and Lemon Streets, Lancaster, Pa., for United States Institute for Textile Research, Inc., Boston, Mass.

Subscription price $5.00 a year. Single copies .50.

Annual Meeting Program

A SCIENTIFIC magazine cannot be held for last-minute news flashes as can a daily paper, therefore at this writing (Oct. 15th) only a rough outline of the research symposium of our annual meeting, that will follow the luncheon-meeting starting at 12:30 P. M., Thursday, Nov. 2, can be given. It will be held in the West Ball Room of the Hotel Commodore, New York City, and both the formal annual meeting of the Corporation and the symposium are planned to be as brief and informative as possible.

The symposium on The Status and Correlation of Current Textile Research in the United States will be introduced and directed by Dr. Vannevar Bush, Vice-President and Dean of Engineering of Massachusetts Institute of Technology. At least two important completed pieces of textile research will be described and their practical application discussed. One will be found of equal interest to all textile manufacturers; another will be of particular importance to the wool industry. Among those who have promised to lead the discussion are Dr. H. DeWitt Smith, an authority on fibre research and colloidal chemistry, and Ephraim Freedman, director of the R. H. Macy Company's laboratory. It is also expected that a representative of the Shirley Laboratory of the British Cotton Industry Research Association, who is now in this country, will take part in the discussion.

Hon. Francis P. Garvan, President of U. S. Institute, will preside at the annual meeting, and Franklin W. Hobbs, Chairman of The Textile Foundation, has promised to outline the present organization and functioning of that research body.

In view of the fact that, after three years of promotional and organization work, it is now possible to speak of research accomplishments, instead merely of research plans, this meeting should attract the attendance of all connected with the industry who appreciate the value and need of new scientific textile knowledge.

Influence of Protein Constituents on Wool Fibres

Part 1.—Survey of Literature; "Elasticum: The Carbohydrate of Wool"; Composition of Protein Constituents of Wool

By WILBER O. TEETERS*

Survey of the Literature

IN the following general survey of the investigations concerning wool an attempt has been made to cover completely all of the essential material in order to obtain a clear conception of the type and amount of work that has been carried out in this field. Those particular articles were chosen which seemed to throw some light on the present knowledge of the chemical properties and structure of the molecule or molecules comprising wool. The first portion, on the physical constitution of wool, serves merely as an introduction to its chemical constitution and properties, while the latter part deals not only with wool but also with the fundamental units making up a protein molecule, namely the amino acids and peptides. Their characteristic and essential reactions are outlined.

Physical Structure of Wool

The woolly, hair-like covering of the sheep forms the most important and the most typical of the textile fibres which are obtained from the skin tissues of different animals. Sheep in their natural condition produce two kinds of hair: the one giving a long, stiff fibre, which is called the "beard-hair"; and the other a shorter, softer, and more curly fibre, which has been named "wool-hair," or true wool. By domestication and proper breeding the sheep can be made to produce the latter kind of hair

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almost exclusively, with very little or none of the hairy fibre. It is this fibre that is of such extreme importance in the textile industry and the one with which the present report is concerned.

Wool, in common with all kinds of hair, is a growth originating in the skin or cuticle of the vertebrate animals, and is an organized structure growing from a root situated in the dermis or middle layer of the skin. Its ultimate physical elements are several series of animal cells of different forms and properties. The root of the wool fibre is termed the hair follicle; it is a gland, which secretes a lymph-like liquid, from which the hair is gradually developed by the process of growth. The hair follicle also secretes an oil which is supplied to the fibre during its growth and serves the purpose of lubricating its several parts, giving it pliability and elasticity. Sebaceous skin glands secrete a fatty or waxy substance, commonly known as wool fat, which serves to coat the surface of the wool fibre. This coating preserves the fibre from mechanical injury during its growth and also prevents the fibres from becoming matted and felted together.

There is also a wool oil which is contained in the cells of the fibre itself and is a true constituent of the latter. This oil should not be removed in the process of scouring (which removes wool fat) because its removal causes the fibre to lose much of its elasticity and resiliency. Wool oil amounts to about 1 per cent of the total weight of the crude fibre whereas the external fatty material amounts on an average to about 8 per cent.[1]

Morphologically considered the wool fibre consists of three distinct portions:

(*a*) In the center of each fibre is a cellular marrow, or medulla, which frequently contains more or less pigment matter and to which the wool owes its color. The size varies considerably in different varieties and grades of wool and serves the purpose of providing the living fibre with an inner canal for the flow of juices necessary for its growth. It also adds to the porosity of the fibre.

(*b*) A layer of cellulose fibrous substance, or *cortical tissue,* surrounds the medulla and forms the major constituent of wool. It gives to the fibre its strength and elasticity.

(*c*) An outer layer, or epidermis, covers the fibrous layer which imparts the gloss or luster to the fibre. It consists of flattened cells or scales the ends of which generally overlap each other and project outwards, causing the edge of the fibre to present a serrated appearance. This scaly covering gives the fibre its quality of rigidity and resistance to crushing strain.

FIG. 1

FIG. 2

Figure 1 shows the diagrammatic cross section of a typical wool fibre while Figure 2 shows a longitudinal section of the same fibre. A, medulla; B, cortical or fibrous layer; C, epidermal or scaly layer.

Chemical Composition of Wool

In its chemical constitution wool is closely allied to hair, horn, feathers, and other epidermal tissues. A distinction must be made between the fibre proper and the raw fleece as it comes from the sheep. In the latter condition it contains a large amount of dirt, grease, and dried up sweat (suint) which must be removed by a scouring process before the pure fibre is obtained. In order to show the average amount of pure fibre which can be obtained from raw fleece wool, the following analysis by Chevreul of a merino wool is given: (These figures are based on wool dried at 100° C.)

	Per Cent
Earthy matter deposited by washing the wool in water	26.06
Suint or yolk soluble in cold distilled water	32.74
Neutral fats soluble in ether	8.57
Earthy matters adhering to the fat	1.40
Wool fibre	31.23

Calculated on the wet basis (14% moisture) these figures show only about 27.5% of pure wool fibre. The figures in the above table vary widely with different samples of wool, but do

give an estimate of the actual pure wool fibre present in the raw fleece wool as it comes from the sheep.

Chemically the pure wool fibre is a protein, known as keratin. Since its constituents do not appear to be rigidly constant in their proportions, a definite chemical formula cannot be assigned. However, its average composition is as follows:[1]

Carbon	50%
Hydrogen	7%
Oxygen	22–26%
Nitrogen	15–17%
Sulfur	2– 4%

At the present time wool is thought to have a much more definite chemical composition than was supposed a decade ago. For example, it has been shown conclusively by Barritt, who carried out extensive work on the nitrogen content of many different wools, that the variation is not as large as represented above. Likewise, the figures for the sulfur content of different wools have narrowed considerably and most investigators agree within 0.3–0.5% sulfur.

The amino acids obtained by the degradation of wool have been studied for some time, the percentage of each present in the wool varying with different investigators. The primary reason for some of the large variations is due probably to the inadequate methods available for complete separation of the individual amino acids and their determination without any loss. One of the first analyses was reported by Abderhalden and Voitinovici[2] in 1907. Ten amino acids were listed and the following percentages of each stated to be present in wool:

	Per Cent		Per Cent
Glutamic acid	12.9	Tyrosine	2.9
Leucine	11.5	Valine	2.8
Cystine	7.3	Aspartic acid	2.3
Alanine	4.4	Glycine	0.58
Proline	4.4	Serine	0.1

Many of the values are, perhaps, wrong, but all are listed since this is the only table showing the percentages of certain amino acids. In 1929 accurate analyses on the chemical composition of wool were carried out by H. R. Marston.[3] In the following table the first three values are considered quite accurate and the remainder approximations only:

1. Cystine	13.1%	7. Amide Nitrogen	1.2%
2. Tyrosine	4.8%	8. Sulfur	3.58%
3. Tryptophane	1.8%	9. Total Nitrogen	17.8%
4. Arginine	10.2%	Isoelectric point estimated pH, 3.4	
5. Histidine	6.9%		
6. Lysine	2.8%		

H. B. Vickery and R. J. Block,[4] however, redetermined the values of the basic amino acids and suggested that Marston had been led to an erroneous conclusion with respect to histidine yielded by wool. Furthermore, Argiris, Stewart and Rimington,[5] have attempted the determination of histidine in tissues of this type and invariably report very low proportions of this amino acid as shown by the following data:

	Vickery and Block	Stewart and Rimington
Histidine	0.66%	0.55%
Arginine	7.8%	6.0%
Lysine	2.3%	2.2%

In all, there have been found 14 amino acids present in the decomposition products of wool: tryptophane, and the three basic amino acids previously noted, in addition to those given by Abderhalden and Voitinovici.

Sulfur seems to be one of the most variable elements of wool. Values for the percentage of sulfur present in wool vary in the literature from 0.8 per cent[1] to 4.52 per cent.[6] However, the most numerous articles report a range of 3.24% sulfur to 3.58% sulfur. The nature and the condition of sulfur in wool have not been fully determined although Rimington states that the sulfur groupings are less reactive than would be expected from cystine peptides and anhydrides. Free –S–S– or –S–H groups are not present in undamaged wool. Salomone,[7] as the result of his investigations, has been led to the conclusion that there are two different sulfurized protein complexes. In one, the sulfur complex is easily decomposed by weak alkalies and oxidizing agents, while in the other the sulfur is very stable and only detached when total decomposition of wool is brought about by heating with concentrated nitric acid.

It has also been shown that wool exposed for a long period of time to ultra violet rays becomes slightly acid due to the formation of sulfuric acid, indicating that the sulfur atoms in wool

are not all similarly combined. Salomone has found that the sulfur containing compounds isolated from the decomposition of wool by acids or alkalies include thio-lactic acid, thio-glycol, ethyl sulfide, methyl and ethyl mercaptans, hydrogen sulfide, cystine and cysteine (cystine predominating). There has been considerable controversy as to the percentage of sulfur in wool that is recoverable as cystine. Barritt [8] and King [9] claim that only 66% of the sulfur present in wool is in the form of cystine, while Rimington [10] and Marston [11] have shown that 13.1% of the wool is cystine which corresponds to 3.50% sulfur. This is approximately the amount of sulfur present in wool, or, in other words, wool is capable of yielding its entire sulfur content as cystine when hydrolyzed by acids.

Wool contains both aliphatic and aromatic amino groups, and, since the total amino nitrogen is greater than that lost by deamination, it seems probable that they may be present in the form of imino groups. Trotman and Wyche [12] have reported the total amino nitrogen as probably about .7%, while, according to Marston's work,[11] the amide nitrogen is 1.2%. The properties of ordinary and deaminated wool were studied extensively by Trotman and Wyche [12] and they find that both have the same shrinkage, elasticity, and tensile strength. As in the case of sulfur, the literature reports large variations in percentage of nitrogen present in different wools. Bowman [13] gives values ranging from 17.8% *N* up to 19.1% *N*. Trotman [14] found from 15.7% to 16.94%, and Gortner [15] obtained 16.27% *N* on white wool and 15.11% for the black from the same animal. Barritt,[16] however, carried out conclusive and valuable work on the percentage of nitrogen present in wool. He used 36 different wools and except for the colored wools obtained consistent values between 16.50% *N* and 17.07% *N* calculated on a dry basis.

The Action of Certain Reagents on Wool

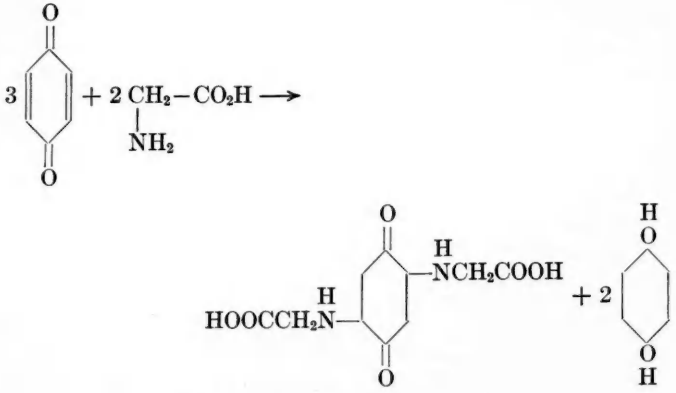
Before leaving the subject of the general chemical constitution of wool and going to the various individual researches carried on in an endeavor to throw further light on the chemical structure of wool, some discussion should be given concerning the action of a few of the more common chemical agents on wool. In its chemical reactions wool is amphoteric in nature;

that is, it exhibits the characteristics both of an acid and a base. The acid nature accounts for the possibility of the formation of compounds of the fibre with various metallic salts, alkalies, and metallic oxides. The coefficient of acidity (number of milligrams of caustic potash neutralized by one gram of substance) of wool is 57. When treated with dilute acids the wool fibre does not appear to undergo any appreciable change; although, from the fact that acids are very readily absorbed by wool and very tenaciously held by it, there is reason to believe that some chemical combination takes place between the fibre and the acid. Fort and Lloyd [17] came to the conclusion that some acid was retained permanently by the wool fibre, but later Harrison [18] showed that after 24 consecutive washings all of the acid had been removed and consequently there was no evidence of any chemical combination between the fibre and the acid. Although very resistant to the action of acids, wool is very sensitive to alkalies, so much so, in fact, that a 5% solution of caustic soda will completely dissolve wool in a few minutes. Concentrated alkali (30–38%) has a peculiar action on wool, increasing the tensile strength of the wool 25 to 30%. Addition of glycerol renders the action of the alkali more effective. The most satisfactory explanation of the nature of the action is that the additional tensile strength is accounted for by the closer adhesions of the scales, giving a greater rigidity to the fibre.

Reducing agents in general have no action on the wool fibre itself, though they reduce the coloring matter in wool and consequently are useful as bleaching agents. Oxidizing agents in dilute solutions act as bleaches, while strong oxidizing agents in concentrated solutions greatly deteriorate wool in its qualities. Toward chlorine wool acts in a peculiar manner; it is completely decomposed by moist chlorine gas, but in dilute solutions of hypochlorites it absorbs a considerable amount of chlorine and becomes harsh, has a high luster and loses its felting properties. The chemical action of the chlorine (bromine acts similarly) on wool is evidently one of oxidation rather than a combination of the fibre with the chlorine. The increased luster and the loss in felting properties seem to be due to the softening and fusing together of the free protruding edges of the scales, which also undergo partial decomposition.

Trotman and Trotman [19] have studied the action of hypochlorous acid on wool and have found that the carbonyl group of the wool is altered when treated with hypochlorous acid, while chlorine itself does not alter the carbonyl group in wool. The action of alkali on wool, as obtained in an alkaline scouring bath, is greatly decreased if formaldehyde has been added to the bath (¼ to $\frac{1}{10}$ of 1%). At one time this action was thought to be a catalytic one. However, it has been shown that there is a direct chemical union of the wool with the formaldehyde, since the addition of acid liberates formaldehyde.

Cooper and Nicholas [20] have studied the germicidal action of paraquinones and their reaction with proteins in general. The paraquinones reacted with proteins (absorption of quinones by proteins) and also with amino acids. Since the quinones react much more rapidly with amino acids than with the complex protein molecule, germicidal effect appears to be due to the reaction of quinones with the simple units instead of the protein molecule as a whole. The reaction appears to be of the following type:



The Protein Molecules of Wool

The protein molecule or molecules which make up keratin have been studied by comparatively few investigators. However, in the past few years there has been considerable research

in an effort to obtain a more intimate knowledge about the chemical structure of the keratin in wool. Speakman and his colleagues have made use of x-rays to determine the arrangement of the molecules in the keratin complex. Their work is based upon the interpretations of the photographs obtained and is mainly of theoretical nature. Considerable data has been collected and the knowledge of the chemical structure of wool broadened by degradation of the wool by use of acids, alkalies, and sodium sulfide solutions, and subsequent analysis of the partially degraded products. The best picture of what is known at the present time about wool chemically can most satisfactorily be shown by a fairly thorough review of the work of the individual investigators.

It has already been shown that there is a slight variation in the amounts of some of the elements present in wool. If wool protein is an absolutely definite chemical compound there would be no such noticeable differences obtained upon analysis of different wools. Also Trotman, Trotman and Sutton, and Küster and Köppel state emphatically that their investigations point to the presence of at least two and possibly more distinct protein molecules in wool protein.

Trotman, Trotman and Sutton [21] studied the action of acids and alkalies on the wool fibre. Cold dilute alkali solutions appear to attack the cortex more readily than the epithelium. An interesting table on the amounts of sulfur removed in 12 hours by varying concentrations of cold sodium hydroxide was submitted with their report: 1. *N*, 50% *S;* N/5, 50% *S;* N/10, 23.6% *S;* N/50, 20.0% *S;* and N/100, 0% *S*. When wool was soaked with 1. *N* sodium hydroxide for 24 hours, the solution filtered and the filtrate acidified, a copious precipitate was obtained which gave protein reactions and contained 1.72% sulfur. Solution and reprecipitation gave a compound which contained 0.9% sulfur. No further loss of sulfur on repeating this procedure could be obtained. Upon treatment of wool with concentrated hydrochloric acid in the cold no hydrogen sulfide could be detected coming off, but the protein passed gradually into solution and it appeared that the dissolved protein was chiefly that of the cortex, since after 48 hours the epithelial scales were still intact. With hot hydrochloric acid there was

also a sparingly soluble residue. The investigators obtained two products (protein A and protein B) in the following manner: Wool was treated with concentrated hydrochloric acid until most of it had dissolved. The solution was filtered, the filtrate neutralized and again filtered. The precipitate was dissolved in ammonium hydroxide and acidified with acetic acid. This precipitate was suspended in water and dialyzed in order to free it from inorganic salts. The residue was coagulated by alcohol, filtered and dried. This material was called protein A and gave the following analysis: Ash, .40%; sulfur, 3.62%; nitrogen, 12.5%. Protein B was obtained from the undissolved residue from the hydrochloric acid treatment by dissolving it in dilute ammonium hydroxide, filtering, neutralizing with acetic acid, dialyzed free from salts and dried. Its analysis gave: Ash, 0.10%; sulfur, 1.49%; nitrogen, 12.68%. It has been considered possible by these workers that the protein B is chiefly present in the cortex while protein A is more abundant in the epithelial scales. The presence of a third protein was indicated by the fact that upon the addition of magnesium sulfate to the filtrate from which protein A was separated, a precipitate was obtained which analyzed 4.05% sulfur. When protein A was treated with chlorine considerable sulfur was lost, whereas protein B lost none under similar conditions. Protein A was more susceptible to oxidizing agents than protein B. Both gave characteristic protein reactions. In conclusion, Trotman, Trotman and Sutton find the great difficulty lies in the complexity of the molecule and are firmly of the belief that there are at least two or more proteins in wool keratin.

One of the most valuable studies of the stepwise degradation of wool, in an attempt to throw some light on its structure, was that by Küster and Köppel in 1927 and later by Küster and Irion in 1929. The wool was degraded by the use of a 3% solution of sodium sulfide which undergoes successive oxidation and reduction: $Na_2S \rightarrow NaSH \rightleftharpoons Na_2S_2$. At the same time the organic substrate also undergoes oxidation and reduction. Sodium thiosulfate and sodium sulfate were actually isolated from the reaction mixture.

Küster and Köppel's [22] work is quite interesting, since it shows the possibilities of the stepwise method of the degrada-

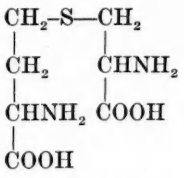
tion of wool. As the wool dissolves in the sodium sulfide, part of the original keratin molecule undergoes disintegration with the formation of ammonia and diffusible nitrogenous cleavage products. The remainder of the wool yields at least two proteins which are also called A and B. Protein A dissolves in 3% sodium sulfide after 125 hours, but protein B is still mostly insoluble. The matter which is in solution as the sodium salt is about one-half the original substance. After only five hours treatment with 3% sodium sulfide the insoluble residue amounts to about two-thirds of the original substance. The basic amino acids are most readily broken off while the mono amino acids and dicarboxylic acids are more resistant. Of the two hypothetical proteins of the wool, the one which goes into solution first (A) is probably richer in basic components. It probably contains a nucleus, however, which resembles in chemical constitution the more stable protein B. The analysis of protein B after hydrolysis with hydrochloric acid was as follows: Glycine, alanine, valine, leucine, aspartic acid, glutamic acid, proline and a residue containing leucine anhydride. It was found that if the acetic acid precipitate, obtained from the solution of wool which has been in contact with 3% sodium sulfide for one hour, is redissolved or left in contact with sodium sulfide solution for three 4-hour treatments (that is, reprecipitating the dissolved material with acetic acid after each 4-hour treatment) a protein results which remains constant in nitrogen content. This protein is identical, as far as per cent nitrogen is concerned, with the one they obtain upon precipitation after 125 hour contact of wool with 3% sodium sulfide. This is the compound called protein B. It analyzed such that the resultant values are in good accord with a formula they suggest:

	Carbon	Hydrogen	Nitrogen	Sulfur
	%	%	%	%
$C_{34}H_{59}O_{12}N_7S$ Calculated	51.68	7.53	12.42	4.06
Mol. wt., 789.77; experimental values;				
12 hrs. ppt.	51.04	7.47	12.8	3.79
125 hrs. ppt.	50.62	7.38	12.8	3.96

Upon methylation three methyls added. Per cent CH_3, 3.04. Calcd. for 3 methyls, 2.85%.

In 1929 Küster and Irion [23] carried out more work on the preparation of the acetic acid precipitate in fairly large amounts.

The analysis of a series of precipitates obtained by allowing wool to remain in sodium sulfide solution over a period ranging from 2½ hours up to 480 hours was reported. Their attention was then turned to the more fundamental units, namely, the amino acids, peptides, diketopiperazines, and azlactones. The low histidine content in the acetic acid precipitate was not due to the destruction of histidine during hydrolysis, since histidine and various derivatives thereof are stable toward sodium sulfide. Likewise arginine, tyrosine and cystine yield no ammonia when treated with sodium sulfide, but the amide nitrogen was split off quantitatively from asparagine. Hence the ammonia liberated from wool by sodium sulfide probably represents amide nitrogen. Cystine is undoubtedly the amino acid most susceptible to the action of sodium sulfide, since the sulfur bridge presents a conspicuous point of attack for reducing agents. The keratin molecule is thus ruptured at each cystine linkage by an opening of the sulfur bridge with the formation of cysteine. The size of the fragments would depend on the distance between the cystine groupings in the peptide chain. The larger and less diffusible fragments should have the lowest sulfur content. The acetic acid precipitate contained more sulfur than the wool itself, which need not be surprising in view of the reactivity of the terminal cysteine, especially when oxidative influences are present. From the acid hydrolysate no cystine or cysteine could be detected. Finally there was obtained a crystalline substance which was more stable to alkalies and more reactive to potassium permanganate and contained only half as much sulfur in the molecule as cystine. Analysis and chemical tests showed that the investigators were dealing with a dicarboxylic acid containing one sulfur atom and giving the formula $C_7H_{14}O_4N_2S$. It was suggested that the compound was probably:



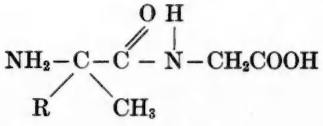
Its copper salt was isolated and analyzed.

It has long been a question as to whether or not there are substituted diketopiperazines present as such in the protein molecule. Most of the evidence at the present time leads one to believe that the diketopiperazine ring is not a normal form. Levene and Bass [24] studied rates of racemization and found that diketopiperazines had the characteristic property of rapid racemization. In the presence of 0.1 *N* to 0.2 *N* alkali about 80% was racemized while strong alkali (0.5 *N* to 1.0 *N*) produced rapid hydrolysis to the corresponding dipeptides and very little racemization. It seemed that, by the application of this procedure, information could be obtained as to the presence or absence of diketopiperazines in the protein molecule. It was found that proteins did undergo racemization to equilibrium which is what would be expected if diketopiperazines were present. However, the proteins only racemized to an extent of 15%, while if diketopiperazines were present there should have been at least 72% racemization. Later, it was found that tetrapeptides under the same conditions racemize which heretofore had been unknown. Thus this work casts doubt on the value of racemization studies for detecting diketopiperazines.

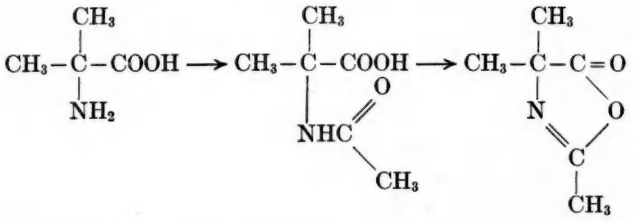
H. D. Dakin [25] made use of the fact that aromatic aldehydes condense with glycine anhydride (diketopiperazine) with the elimination of water. If a protein known to contain a large amount of glycine was treated with benzaldehyde, phenyl alanine should be present in the hydrolyzed product. The reaction proceeded smoothly but the product obtained was extraordinarily resistant to hydrolysis. No phenyl alanine was found and the same products were obtained from proteins poor in glycine. This work, therefore, is somewhat in favor of the theory that diketopiperazine rings are not present normally in the protein molecules.

The remainder of this review will contain a brief account of some of the researches that have been carried on with amino acids and peptides. H. D. Dakin and R. West [26] observed a peculiar reaction when amino acids were treated with acetyl chloride and pyridine.[27] Substituted acetylated amino acetones were the products obtained. A mechanism for the reaction was suggested. Levene and Steiger [28] made the same observation but suggested a somewhat different mechanism.

In 1931 Levene and Steiger [29] prepared a series of dipeptides of the type,



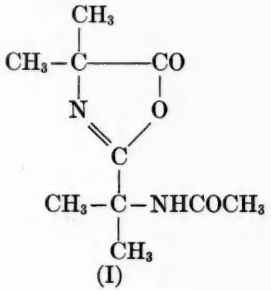
and studied the action of acetic anhydride on tertiary amino acids and dipeptides. Experiments showed that bromacyl glycines are aminated at an exceedingly low rate and that isolation is difficult. It seemed necessary to use the azlactone method of synthesis developed by Moler and modified by Bergmann. No difficulties were met in the case of phenyl methyl amino acetic acid and the azlactone was obtained in good yields. However, in the case of analogous compound, α-amino isobutyric acid and *dl*-isovaline and azlactone could not be isolated. α-Amino isobutyric acid boiled with acetic anhydride brought about polymers, but when the amino acid was repurified a clear solution resulted. Upon removal of the acetic anhydride and acetic acid an oily residue remained which was found to be a lactone $C_{10}H_{16}O_3N_2$. From pure *dl*-isovaline analogous results were obtained. Steps leading to this lactone may best be represented by the equations:



Acetyl dipeptide ($C_{10}H_{18}O_4N_2$) $\xrightarrow{-H_2O}$ Lactone $C_{10}H_{16}O_3N_2$

Three possible structures are given for the lactone. In water at 25° it is hydrolyzed to N-acetyl α-amino isobutyryl-α-amino isobutyric acid. A surprising thing happens in water at 100° in that N-acetyl α-amino isobutyric and α-amino isobutyric acid are obtained. It is worthy of note that α-amino isobutyryl-α-

amino isobutyric acid is stable to water, thus indicating a sharply marked case of substitutional activation. The investigators showed that the degree of instability of the peptide linkage depends considerably upon the structure of the first amino acid. N-Acetyl glycyl α-amino isobutyric acid only hydrolyzed 10% in four hours, while N-acetyl amino isobutyryl-α-amino isobutyric acid is hydrolyzed 90%. Hence, it was concluded that acetic anhydride converts tertiary amino acids, such as α-amino isobutyric acid or *dl*-isovaline, to the azlactone. The latter then couples with some of the unacetylated amino acid giving a dipeptide which acetylates and loses water to form a lactone, the structure of which is probably one of the following suggested by Levene and Steiger:



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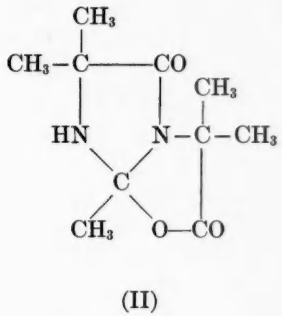
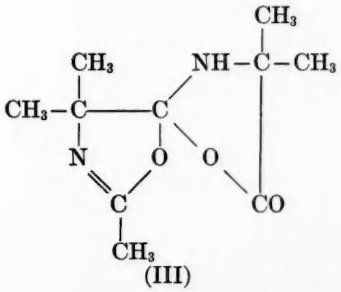


(II)

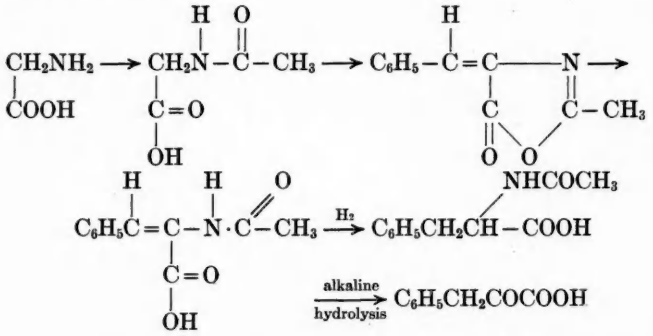


(III)

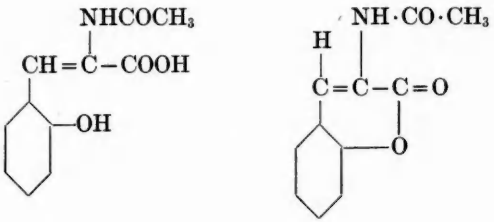
The first (and the only one hitherto known), lactone of a N-acyl dipeptide, has been prepared by Mohr and Stroschein.[30]

Condensation of aromatic aldehydes with acyl derivatives of glycine has, according to Dakin,[31] furnished the most useful

method available for the synthesis of compounds which may be in turn converted into aromatic amino and ketonic acids, such as tyrosine, tryptophane and thyroxine. By first acetylating glycine, a better yield is obtained, since there are two competing reactions which lower the yield considerably. The reactions indicated above are



This method for preparing alpha keto acids is of great value, according to Dakin, as the difficulties of Erlenmeyer's method and others are entirely avoided. Good yields and products easily separated are obtained. An unusual reaction was observed by using salicylaldehyde, since α-acetamino coumarin was obtained instead of the expected o-hydroxy α-acetamino cinnamic acid. The change from one to the other can be represented as follows:



The Carbohydrate of Wool

One compound of great interest found in wool, which is not a protein, is a carbohydrate occurring between the external

plate-cells (scales) and fibre cells (body of the fibre). This carbohydrate was called "elasticum" by Allwörden.[32] The presence of elasticum was based on the formation of a precipitate, supposed to be an osazone,[32] as the result of treating a concentrated aqueous extract of wool with phenylhydrazine. In 1922 H. Lagache [33] also reported the isolation of an osazone from a carbohydrate in wool which had the same melting point as that of galactosazone. However, Trotman, Trotman and Sutton [34] in 1926 were unable to detect the presence of any carbohydrate in wool and could not obtain the osazone described by previous workers.

Allwörden [32] attributed many of the peculiar and valuable properties of wool to the presence of this carbohydrate among the scales of the wool filaments. It has been suggested that if this substance is removed (soluble in dilute alkalies), the fibres are no longer capable of being felted or finished properly, and are brittle and weak.

Elasticum: The Carbohydrate of Wool *

THE presence of a carbohydrate, "Elasticum" on wool fibres was reported in 1916 by Allwörden [32] who attributed many of the peculiar and valuable properties of wool to the presence of this carbohydrate among the scales of the wool filaments. It was supposed to be connected with the swelling and fulling properties of wool. Because of the technical importance of these properties a further study of this unique carbohydrate seemed desirable.

The presence of elasticum was based on the formation of a precipitate, supposed to be an osazone,[32] as the result of treating a concentrated aqueous extract of wool with phenylhydrazine. In 1922 H. Lagache [33] also reported the isolation of an osazone from a carbohydrate in wool which had the same melting point as that of galactosazone. However, Trotman, Trotman and Sutton,[34] in 1926, were unable to detect the presence of any carbohydrate in wool and could not obtain the osazone described by previous workers.

In the present investigation New Zealand wool was carefully freed from all foreign matter by hand picking and then subjected to the procedures outlined by Allwörden [32] and by Lagache.[33] Since these gave negative results other extraction methods were tried. The typical procedure followed in all the experiments was as follows:

About 500 g. of New Zealand wool was placed in an earthenware crock and covered with 8 l. of the extracting solutions. After standing 24 hours

* A study by Mr. Teeters published previously by permission of The Textile Foundation.

The author was indebted to Dr. E. A. Rees of the Kenwood Mills for his kindness in supplying the wool used in this investigation.

the wool was removed, squeezed as dry as possible and again extracted with 8 l. of the solution. The combined extracts were then placed in parchment bags and subjected to dialysis, in order to separate the corbohydrate from any proteins or their degradation products. The dialysates were combined and concentrated by distillation *in vacuo*. The concentrated solutions were clarified by treatment with infusorial earth and filtered. This filtrate was then subjected to a series of tests for monosaccharides, in order to detect the presence of any carbohydrate.

This procedure was carried out on (1) raw wool, (2) wool washed with distilled water and (3) wool scrubbed by extraction with petroleum ether. The extracting solutions were as follows:

1. Distilled water.
2. Dilute sodium hydroxide solution (2%) (Method of Allwörden).
3. Dilute sodium carbonate solution (0.2%) (Method of Lagache).
4. Dilute hydrochloric acid solution (2%).

The results of these treatments and tests are summarized in Table I.

Table I

Tests for Carbohydrates in Extracts of Wool

Test	Extracting Solutions				
	Water	NaOH	Na_2CO_3	HCl	Galactose control
Molisch	+	+	+	+	+
Fehling's	−	−	−	−	+
Bareford's	−	−	−	−	+
Benedict's	−	−	−	−	+
Phloroglucinol-HCl	−	−	−	−	+
Phenylhydrazine	−	−	−	−	+

The only positive tests in Table 1 were obtained with the Molisch reagent. Since all other tests were negative, and since the Molisch test is given by compounds other than carbohydrates, the data indicate that there is no carbohydrate present in this New Zealand wool.

In order to determine whether a carbohydrate could be detected by the above procedure .25 g. of gelactose (the sugar suggested by Allwörden and Lagache) was added to 500 g. of wool and the above procedures repeated. In every case, except that in which 2% sodium hydroxide solution was used as the extracting medium, all the tests for galactose were positive. In the run using 2% sodium hydroxide the tests were negative, indicating that the strong alkali had degraded the galactose.

The present work indicates, therefore, that this New Zealand wool does not contain any carbohydrate, and, hence, that "Elasticum" does not play any part in the properties of such New Zealand wool fibres.[35]

The formation of a precipitate with phenylhydrazine, observed by other workers, might have been due to the degradation of the proteins by alkalies. All of the extracts of New Zealand wool gave positive biuret

tests, indicating that some of the low molecular weight peptides had been formed and dialyzed through the parchment.

Summary

No carbohydrate which would correspond to the previously reported "Elasticum" of wool could be detected in New Zealand wool. The changes involved in its felting or fulling processes must, hence, be concerned with changes in the protein constituents of the fibres.

Composition of the Protein Constituents of Wool

In order to determine the constitution of keratin, the protein of wool, several methods of degradation have been applied by previous investigators. These are:

1. Acid hydrolysis
2. Alkaline hydrolysis
3. Sodium sulfide degradation

Complete acid hydrolysis leads exclusively to a mixture of α-amino acids. Many of these have been identified and the amount present determined. Table I shows the percentages of the various amino acids present in wool.

Table I

Amino Acids Comprising Wool Keratin

	Per Cent
1. Cystine	13.1
2. Glutamic Acid	12.9
3. Leucine	11.5
4. Arginine	6.0
5. Tyrosine	4.8
6. Alanine	4.4
7. Proline	4.4
8. Valine	2.8
9. Lysine	2.3
10. Aspartic Acid	2.3
11. Tryptophane	1.8
12. Histidine	0.66
13. Glycine	0.58
14. Serine	0.10

Partial acid or alkaline hydrolysis indicates that two different proteins are present in wool: Protein A, in the epithelial cells

and Protein B in the cortex. Degradation by means of solutions of sodium sulfide also indicates the presence of two proteins.

The decomposition of certain proteins by means of alcohol has yielded very interesting results. Since this method has not been previously applied to wool, a study of this degradation procedure has been started in the hope of determining at least some of the components of keratin.

Alcoholysis of Proteins

An alcoholic solution saturated with hydrogen chloride has been used as a reagent to hydrolyze and degrade proteins. An attempt was made to shorten the double process of Emil Fischer's for isolating amino acids, by using this reagent from the beginning to serve both as a hydrolysing and esterifying agent. Alcoholic hydrogen chloride is undoubtedly a milder reagent than a saturated aqueous solution of the same: (*a*) because the solubility of hydrogen chloride in alcohol is much less than in water; (b) the solution cannot be heated to as high a temperature.

Pribram [36] studied the applicability of alcoholysis in metabolism research and found evidence that esterification did not take place, but that a small amount of water (which is unavoidably present) hydrolyzed a portion of the protein material to amino acids, and these esterified. This process generated water, which, in turn, hydrolyzed more of the complex protein. Abderhalden and Hanslian [37] found later that alcoholic hydrogen chloride refluxed for six hours was not a vigorous enough treatment to split the peptide linkage. Other investigators have reported that the simple amino acids present were a very small fraction of the degradation products obtained, thus confirming Abderhalden and Hanslian. This fact has led several investigators to make use of this reagent in the study of the chemical composition of protein, since the molecule is degraded to simpler units but not broken down to the fundamental amino acids.

There has been considerable work in the past ten years concerning the internal chemical arrangement of the protein molecule. Sadikov and Zelinskii [38] state that "the constitution of the protein molecule is not that of a polypeptide, as Emil

Fischer proposed, but we are dealing with a system of rings among which the '*peptine*' (diketopiperazine) rings occupy a dominant part.'' In a great many of the more recent works dealing with this subject, the inadequacy of the Fischer polypeptide linking of the amino acids to explain alone the large number of reactions of the proteins, has come to the attention of those interested in the constitution of the protein molecule.[38, 39, 40] In an attempt to throw some light on this question Gränacher, Karrer, and Schlosser [41] degraded goose feathers with neutral alcohol under pressure. A thorough study of the degradation products produced was made and a white crystalline solid was isolated which proved to be a mixture of diketopiperazines. Known polypeptides were treated in a similar manner and it was found that they were not broken down by this treatment, nor were diketopiperazine rings formed. On the other hand, recent studies [42] on polymerization have shown that the amino acids form ''chain'' polymers with great ease.

Experimental Work on Alcoholysis of Wool

A. *Properties and Analysis of the Raw New Zealand Wool Used in These Experiments.*

The results of the analyses of the raw fleece are recorded in Table II, and were obtained by the usual methods of analysis.

Table II

	Per cent
1. Water Insoluble Material	14.1
2. Alcohol Soluble Extract	13.8
3. Moisture (air equilibrium)	7.65
4. Ash	0.1
5. Protein Fibre	64.35
Composition (calculated on a dry, ash-free basis)	
Carbon	50.93
Hydrogen	7.23
Nitrogen	16.39
Sulfur	4.36
Oxygen (by difference)	21.09

B. Degradation by Alcoholic Hydrogen Chloride

1. General Procedure.—Two hundred grams of unscoured New Zealand wool was washed thoroughly with distilled water

and then subjected to extraction with low boiling (60–70° C.) petroleum ether. The clean wool was dried in a vacuum oven for 12 hours at 85° C. When the wool has become thoroughly dry it was immediately placed in 8 l. of absolute ethyl alcohol contained in a 12 l. round-bottomed flask. The flask was placed on a steam cone and connected to another trap, which led to a flask containing 300 cc. of a 3% mercuric chloride solution. The outlet of this flask carried the hydrogen chloride to a water trap. After five days refluxing the original wool had disappeared completely, with the formation of an alcohol insoluble solid (solid product *A*), an alcohol soluble fraction (solution *B*), and a volatile portion (C) which consisted primarily of hydrogen sulfide, mercaptans, and organic sulfides.

2. Solid Product A.—After the precipitate in the flask had settled, the greater portion of the alcoholic solution was decanted. The precipitate was filtered and placed in a vacuum to complete the drying. The solid product *A* weighed 67 grams which represented 52% of the original fibre (calculated on a clean dry basis).

Table III

Chemical Analysis of Solid Product A

	Per cent
1. Carbon	48.57
2. Hydrogen	6.38
3. Nitrogen	13.95
4. Chlorine	6.47
5. Ash	2.0
6. Oxygen (by difference)	24.63
7. Sulfur	Nil.

The calculation of the percentage of each element present was based on an ash-free sample.

The general chemical properties of solid product *A* are summarized in Table IV. It was found to be a hygroscopic light brown powder and to decompose at 274–276° C. (corrected). The chlorine present in solid *A* is known to be ionizable and is probably present as the hydrochloride of the amino groups. It was insoluble in all common organic solvents and formed a colloidal solution with water. Such a solution after filtration was optically active and gave D^{25} –49°.

Table IV

Test	Result	Inference
1. Millon's Test	negative	No tyrosine present
2. Hopkin's Cole	negative	No tryptophane group
3. Xanthroproteie reaction	positive	Phenyl group present
4. Biuret Test	positive	Peptide linkage
5. Ninhydrin Test	positive	Free carboxyl and α-amino group

The Preparation of Ninhydrin.—Ninhydrin (triketohydrindenehydrate) is an expensive reagent used in protein chemistry to detect the presence of a free carboxyl and α-amino group. Since it is not a common reagent it became necessary to prepare some in this laboratory. A sufficient quantity of Ninhydrin has been prepared by an entirely new method of synthesis which is much more rapid than that described in the literature.

Constituents of Solid Product A.—Solid *A* was hydrolyzed and the various amino acids present in the molecule isolated. A modification of the method of Brazier [43] for the separation of the products of protein hydrolysis was followed. The general scheme is shown in Chart I.

In order that accurate and dependable results might be obtained by this method it has become apparent that considerable starting material should be used. Twelve and one-half grams of solid *A* was hydrolyzed and several of the various amino acids isolated. The analyses of these acids have not been completed at the present time and, hence, the results will be reported later.

3. Solution B. (soluble fraction).—The filtrate from solid *A* (solution *B*) was a deep brown solution containing approximately 48% of the original weight of the wool fibre used. It was vacuum-distilled to a volume of 250 cc. (thick syrup) and placed in a vacuum desiccator over solid potassium hydroxide for three days. The syrup was divided into three portions and each portion diluted to three times its original volume with water. This diluted solution was cooled to 0° C. by means of an ice and salt bath, and 200 cc. of ether added. Solid sodium carbonate was added as fast as possible (effervescence takes place), until the solution was alkaline. After extracting the water layer with ether, considerable sodium carbonate was added in order to salt out any ethyl esters that might be present, and the ether extraction repeated 10 times. The ether was vacuum-

distilled at 15° C., 25 mm. pressure. The residue should contain the amino acid esters. The water layer was neutralized and evaporated to dryness. On addition of water an insoluble product was obtained. It was filtered, dried, and on analysis yielded the following figures:

C	52.74
H	6.08
S	11.50
Cl	2.17
Ash	3.10
N	No data

The study of this compound is being continued.

Present Status of the Degradation of Wool by Alcoholysis

Clean dry wool has been degraded by using absolute ethyl alcohol saturated with hydrogen chloride. This reagent disintegrated the wool after a five to seven day treatment to such an extent that none of the original fibre was left when the process was completed. The products isolated in this hydrolysis can be enumerated as follows:

1. Volatile compounds containing sulfur.
2. An alcohol insoluble solid containing no sulfur, but giving characteristic protein reactions. Determination of the composition of this compound is now under way.
3. An alcohol soluble fraction composed of (a) an ether soluble portion (probably the ethyl esters of amino acids); (b) an ether insoluble portion from which a compound containing a high percentage of sulfur was isolated.

Summary

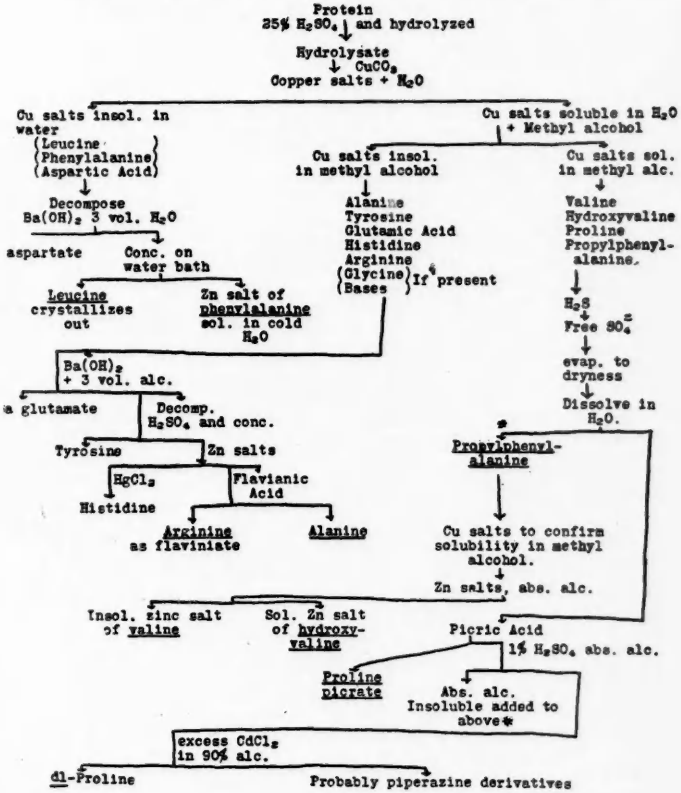
1. A survey of the literature on the chemical composition of wool is presented.

2. A thorough investigation indicates that no carbohydrate, previously described as "Elasticum," is present in New Zealand wool. Hence the reactions and changes involved in the felting of New Zealand wool are due to the protein constituents.

3. A new degradation procedure has been applied to wool and a study of the composition of the products started.

CHART I

Method for the Separation of the Products of Protein Hydrolysis.



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The Disposal and Recovery of Textile Wastes

Part I (Concluded).—Disposal by Biological Treatment; Disposal by Superchlorination; Precipitation With Chlorinated Sludge; Recovery; Conclusions; Future Studies

By M. S. CAMPBELL*

B. Disposal by Biological Treatment

FOR the purpose of investigating the possibility of treating the wastes by biological methods, and at the same time determining the toxic effect of the wastes on the biologic life of sewage-treatment plants and streams, a trickling filter has been constructed.

One dimension, the depth, has been built to full scale, and the results obtained will, therefore, be comparable to those obtained in practice.

The filter consists of a 7-in. cast iron pipe, 6½ ft. in length, erected in a vertical position. The filtering medium is of cinders 6 ft. in depth,[13] allowing 6 ins. freeboard. The lower 6 ins. of cinders are screened to 1½–2 ins. in size, and the remaining 5½ ft. are screened to ½–1½ ins. The cinders are supported by a steel plate with 1-in. perforations, spaced as closely as stability would permit. The sewage and wastes are pumped to the top of the filter by use of a centrifugal pump, having a 1-in. discharge, and driven by a ¼ h.p. motor. From the top of the filter the wastes trickle down through the bed of cinders and are collected in a settling basin at the bottom, from which the pump picks them up and returns them to the top of the filter for recirculation. The piping includes the necessary air-cocks and valves for controlling the rate of recirculation, and for aerating the combination of wastes and sewage under pressure.

The filter is of the same construction as that used in the separate sludge-digestion type of sewage treatment plant, but the operation is quite different in order to meet the requirements imposed upon it by the wastes. The departure from the ordinary operation of this type of filter is that the material being treated is recirculated many times through the filtering media. The usual operation is to allow the material to filter through the bed once. The results we are obtaining are encouraging, and prove that the method has many possibilities.

* Mr. Campbell is a Junior Fellow of The Textile Foundation and works under the direction of Dr. H. G. Baity, University of North Carolina.

Theory of Purification by the Trickling Filter

The "Dunbar Absorption Theory of Sewage Purification" is the accepted explanation of the purification performed by trickling filters. According to Dunbar[14] the important factors in the purification are the mechanical action of the filter, adsorption, chemical combination, condensed oxygen, enzymes, microörganisms and higher forms of plant and animal life. Mechanical action, adsorption, and to a limited extent chemical combination, cause the fixation in the filter of the unstable organic matter. The oxygen, enzymes, microörganisms and higher forms of plant and animal life are concerned with the conversion of the retained unstable organic matter to the stabilized mineral form.

The fine pores of the filter are to some extent responsible for the retention of the suspended and colloidal matter, but the majority of retention is due to the slimy bacterial film which is built up on the surfaces of the filtering media in a mature filter.

The bacterial film has an extraordinarily large surface area, and the liquid adhering to the outer surface is under great pressure. Those substances, the solubilities of which increase with pressure, are absorbed by the surface film, all others passing through the filter unabsorbed and unchanged.

Therefore, the mineralized products formed on the outer surface of the film diffuse into the succeeding dose of sewage because they are more soluble at the lower pressure of the sewage.

The absorption would cease with the establishment of equilibrium, but, due to the continuous action on the absorbed substances by the bacterial and biological life in contact with atmospheric oxygen, this condition is never attained.

The atmospheric oxygen is supplied to the filtering media by leaving the bottom of the filter open in order that air may circulate upwards, while the sewage is trickling down through the bed. The oxygen requirements are fulfilled by intermittent dosing of the filter, thus allowing the organic matter adsorbed from one dose to oxidize before adsorption of the matter from the following dose is begun.

It is our belief that, with the proper care of the organisms and operation of this type of filter, a biological life can be developed that will react favorably to textile wastes, and thus convert the unstable organic constituents of the wastes to the stable form, and at the same time destroy all, or a part of, the color.

This belief is supported by the findings of Clark and Adams,[15] who have shown that nitrification ceases when the carbon-to-nitrogen ratio in the sewage becomes too great; but that good purification and the production of a satisfactory effluent can be secured, nevertheless, if the filter is gradually accustomed to such sewage by the application of increasingly larger doses.

Procedure

It will be well to define "rate of filtration," and "rate of recirculation" as employed in the operation of the filter used in these studies. The ordinary operation of a trickling filter is by passing the sewage through it once at the rate of one million gallons per acre per day (M.G.A.D.). This filter is different, however, in that the combined sewage and wastes

are being recirculated through the filter at a rate of several million gallons per acre per day, while the raw wastes and sewage are being added to the circulating material at the customary rate of one million gallons per acre per day, and are drawn off as effluent liquor at the same rate. The rate of filtration as employed here is, therefore, the rate at which the wastes are being added to the circulating material.

The filter was seeded by recirculating settled sewage through it at the rate of 42 M.G.A.D. For the present our findings are qualitative, but the most economical rate of recirculation will be determined after the possibilities of the filter have been determined.

A period of 14 days of recirculation was required before the filter became completely mature. At that time the normal purification data for domestic sewage was obtained to use as a control. The average purification data for the domestic sewage applied to the filter are given in Table 7.

TABLE 7

Normal Purificaion of Domestic Sewage on a Trickling Filter

Rate of Filtration = 1 M.G.A.D.
Rate of Recirculation = 42 M.G.A.D.

Hours of Recirculation	Oxygen Consumed	% Reduction
0	35.8	0.0
1	14.3	60.0
2	9.5	73.5
3	8.0	77.5
4	7.4	79.3
6	6.7	81.3
12	5.2	85.4

It is seen that the maximum amount of purification occurs during the first two hours of recirculation. It would not be economical to circulate sewage longer than that time. It was estimated that for the combined sewage and textile wastes about a 12-hour recirculation period would be required to produce a suitable effluent.

Sulfur Dye Jig Liquor

The filter was dosed with a mixture of 5% sulfur dye jig waste liquor and 95% fresh, settled sewage. The dye liquor was the same sample previously referred to under "Chemical Precipitation," and it will be remembered that no satisfactory results could be obtained by coagulation of this particular waste.

The oxygen consumed, and color determinations were made on the raw sample, and at intervals of 1, 2, 4, and 23 hours during recirculation. The results are given in Table 8.

TABLE 8

Purification of Sulfur Jig Wastes and Sewage on a Trickling Filter

5% Waste, 95% Sewage
Rate of Filtration = 1 M.G.A.D.
Rate of Recirculation = 42 M.G.A.D.

Hours of Recirculation	Oxygen Consumed		Color	
	p.p.m.	% Red.	p.p.m.	% Red.
0	268.0	0.0	550	0.0
1	138.0	48.5	Turbid	——
2	108.5	59.5	Turbid	——
4	90.0	66.4	Turbid	——
23	17.2	93.8	40	92.7

Comparing the results of purification of 5% sulfur jig wastes and 95% sewage with the normal purification of sewage, the purification as determined by the oxygen consumed tests was 11.5% below normal after one hour of recirculation, 14.0% below normal after two hours, and only 11.1% below at the end of four hours. These results indicated that the filter was gradually becoming accustomed to the waste liquor.

At the end of 23 hours the oxygen consumed had been reduced 93.8%, and the color 92.7%. The effluent was clear and sparkling, with no trace of color when held up to the light in a beaker. The color determination indicated a color of 40.

The treatment of the 5% mixture of waste, and 95% sewage was so successful that the fraction of waste was raised to 15%.

Several runs were made with this combination, the results of which appear in Table 9 for Runs 1, 3, 4, 6, 7, and 8. No data were taken on Runs 2 and 5.

That a combination of 15% sulfur jig wastes and 85% sewage can be treated successfully by biological filtration was definitely shown.

The improvement of the effluent of Run 3 over that of Run 1 after 26 hours is shown by the increased reduction in oxygen consumed and color.

After the filter had been in operation for five days the same results were accomplished in 18.5 hours recirculation (Run 4) that previously had required 26 hours (Run 3).

After eight days application of the combined waste and domestic sewage to the filter a satisfactory effluent was obtained with 12 hours recirculation.

An important feature of the operation is the rest period required by the organisms in the filter in order that they might consume the adsorbed organic material. From Run 1 to Run 4 rest periods averaging nine hours were allowed between doses, the filter being operated continuously during runs. The efficiency during these runs was increasing. From Run 4 to Run 8 the filter was operated continuously for four days with only 30 minute rest periods between runs. The runs during this period averaged 16 hours of recirculation.

The effect is reflected in the decrease in efficiency, as shown by the fact that Run 8 required 24 hours of recirculation to produce the same type

TABLE 9

Purification of Sulfur Jig Wastes and Sewage on a Trickling Filter

15% Waste, 85% Sewage
Rate of Filtration = 1 M.G.A.D.
Rate of Recirculation = 42 M.G.A.D.

Run No.	Hours of Recirculation	Oxygen Consumed p.p.m.	Oxygen Consumed % Red.	Color p.p.m.	Color % Red.
1	0	652.0	0.0	1250	0.0
	2.5	458.0	29.8	——	——
	5.0	292.0	55.2	500	60.0
	8.5	274.0	58.0	500	60.0
	22.0	103.0	84.1	200	84.0
	26.0	35.0	94.6	100	92.0
3	0	650.0	0.0	1250	0.0
	26.0	22.0	96.5	70	94.4
4	0	654.5	0.0	1250	0.0
	16.0	28.5	95.8	100	92.0
	18.5	22.0	96.8	100	92.0
6	0	650.0	0.0	1250	0.0
	12.0	38.0	94.1	200	84.0
7	0	652.5	0.0	1250	0.0
	12.0	47.5	92.6	300	76.0
8	0	655.5	0.0	1250	0.0
	24.0	48.0	92.5	300	76.0

of effluent as produced during Run 7 in 12 hours. This result is in direct agreement with "Dunbar's Theory of Purification."[14]

After the filter had become accustomed to the 15% sulfur jig waste concentration the percent reduction in oxygen consumed was higher for the combined wastes than for sewage alone. Recirculation of the combined wastes for 12 hours (Run 6) resulted in a reduction in oxygen consumed of 94.1%, as compared to 85.4% in the same period for domestic sewage as taken from Table 7.

Conclusions:—Treatment of sulfur jig wastes in combination with sewage on a trickling filter is successful.

By gradually increasing the concentration of the sulfur dye waste a satisfactory effluent has been obtained with 12 hours recirculation at a rate of 42 million gallons per acre per day with a concentration of 15% sulfur dye waste and 85% sewage.

It is entirely possible that the concentration of waste can be increased still higher.

The filter requires a daily rest period of several hours. This can be accomplished by running the filter during the working day, and then allowing it to stand over night, care being taken that it does not stand long enough to dry out.

Direct Black Dye Liquor

The waste sample was taken from the Durham Hosiery Mill, Durham, N. C., during the discharge of a direct black dye bath, dyeing cotton hose. The sample is the same as that used in the chemical precipitation tests already referred to under "Chemical Precipitation."

The character of the waste was as follows:

pH	7.2
Total Alkalinity	550 p.p.m.
Pheno. Alkalinity	0 p.p.m.
Color	55,000
Oxygen Consumed	730.0 p.p.m.

The oxygen-consumed-value of this waste is relatively low, which indicates that it could not be expected to respond readily to biological treatment. It was hoped, however, that the biological filtration would destroy the color, since the treatment had been so successful with the sulfur jig wastes. The rate of recirculation was increased to 80 M.G.A.D.

The results of three runs are given in Table 10.

Table 10

Treatment of Direct Dye Liquor on a Trickling Filter

10% Dye Waste, 90% Sewage
Rate of Filtration = 1 M.G.A.D.
Rate of Recirculation = 80 M.G.A.D.

Run No.	Hours of Recirculation	pH	Oxygen Consumed p.p.m.	Oxygen Consumed % Red.	Color p.p.m.	Color % Red.
1	0	7.0	90.5	0.0	5,000	0.0
	21	7.5	14.2	84.3	200	96.0
2	0	7.0	90.5	0.0	5,000	0.0
	25	7.4	14.4	84.1	200	96.0
3	0	7.0	90.5	0.0	5,000	0.0
	7	7.3	40.5	55.2	3,000	60.0
	26	7.4	10.6	88.2	200	96.0

Although the reduction in oxygen consumed was high enough to produce a satisfactory effluent, averaging 13.1 p.p.m. at the end of 24 hours, and averaging a color reduction of 96.0%, the effluent was far from satisfactory, due to the high residual color.

An unexpected development occurred when the effluent from Run No. 3, at the end of a 7-hour recirculation, was passed through an activated carbon filter. During the experiments on the chemical precipitation of this waste, the best effluent produced was filtered through activated carbon, and the lowest color obtained was 750 p.p.m., which is altogether unsatisfactory.

The results of filtration through activated carbon, after biological filtration, are as follows:

Effluent of trickling filter	Color = 2,000
After filtration through carbon	Color = 40

This effluent was clear and sparkling, having the appearance of ordinary drinking water. Recirculation through the biological filter had produced an effluent amenable to color removal by activated carbon.

Whether this effect is due to aeration, or to the biological action of the filter, is yet to be determined.

Conclusions:—Treatment of direct black dye liquor on a trickling filter gives satisfactory reductions in oxygen consumed. It is possible to obtain a reduction of 96% in color, but the residual color is still so high that the effluent cannot be considered satisfactory.

Filtration of the trickling filter effluent through activated carbon produces a clear and sparkling effluent. The work will be continued in order to determine whether the effect of conditioning the waste for color removal by carbon is due to biological action or to aeration.

Kier Liquor

The kier liquor used was obtained from the Erwin Cotton Mills, Durham, N. C. The sample was taken during discharge of the caustic boil, and its characteristics were as follows:

pH	77.5
Total Alkalinity	3,575 p.p.m.
Pheno. Alkalinity	1,855 p.p.m.
Color	10,000
Oxygen Consumed	2,765 p.p.m.

Since the oxygen-consumed of the kier liquor was high, it was expected that satisfactory results could be obtained if the waste was neutralized before application to the filter. It was desirable to neutralize the waste without producing a precipitate, thus avoiding the introduction of a sludge problem. Several methods were tried, and sulphuric acid was found to give the best results.

The results obtained by biological filtration appear in Tables 11 and 12.

TABLE 11

Treatment of Kier Liquor on a Trickling Filter

10% Kier Liquor, pH adjusted to 7.8
90% Settled Sewage
Rate of Filtration = 1 M.G.A.D.
Rate of Recirculation = 42 M.G.A.D.

Hours of		Oxygen Consumed		Color	
Recirculation	pH	p.p.m.	% Red.	p.p.m.	% Red.
0.0	6.8	177.0	0.0	1,000	0.0
16.5	7.5	32.0	81.8	100	90.0
21.5	7.6	28.5	83.9	75	92.5
0.0	6.8	173.0	0.0	1,000	0.0
18.5	7.5	45.0	74.0	75	92.5
25.5	7.7	38.5	77.7	50	95.0
0.0	6.8	160.5	0.0	1,000	0.0
12.5	7.6	48.5	69.8	300	70.0
0.0	6.8	160.5	0.0	1,000	0.0
25.5	7.7	30.5	81.0	400	60.0

The percentage of waste was then increased to 20%, and the rate of recirculation to 80 M.G.A.D. The results under these conditions appear in Table 12.

TABLE 12

Treatment of Kier Liquor on a Trickling Filter

20% Kier Liquor, pH adjusted to 7.8
80% Sewage
Rate of Recirculation = 80 M.G.A.D.

Hours of Recirculation	pH	Oxygen Consumed		Color	
		p.p.m.	% Red.	p.p.m.	% Red.
0.0	7.3	529.0	0.0	2,000	0.0
25.5	7.7	89.5	83.0	750	62.5
45.0	7.8	73.0	86.3	500	75.0

The results obtained indicate that the above method is worth further investigation. It appears that a period of 16 hours recirculation is required for a 10% concentration, the optimum rate of recirculation being 42 M.G.A.D.

The data for the 20% concentration of kier liquor at a recirculation rate of 80 M.G.A.D. is insufficient for determination of the recirculation period.

With higher concentrations of waste the per cent reduction in oxygen consumed is higher, but the residual oxygen consumed is still excessive.

Results obtained with the 10% concentration (Table 11) were very gratifying. The oxygen-consumed reduction is good, as is the color reduction. The effluent is clear, with no suspended matter, and with only a slight tinge of brown color.

A large amount of sludge was produced which settled rapidly, almost filling the lower half of the sedimentation basin before it was observed. This sludge is of an entirely different character than the suspended and colloidal matter in the raw sewage and kier liquor. To all appearances it is stable, but its stability as well as its ease of handling will have to be experimentally determined.

Treatment of the effluent by activated carbon did not reduce the color enough to warrant further investigation.

It is quite possible that the percentage of waste for treatment on the filter can be increased by gradually habituating the biological life to increasing concentration.

Conclusions:—Treatment of 10% kier liquor, neutralized with acid, and 90% sewage on a trickling filter, at the rate of recirculation of 42 M.G.A.D., will produce a good, clear effluent with 16-hours recirculation.

A concentration of 20% liquor has been run, but not continued long enough for conclusive results.

It is our opinion that a satisfactory effluent may be developed with concentrations of kier liquor above 20%.

C. DISPOSAL BY SUPERCHLORINATION

Chlorination of the wastes offered two possibilities: first, that precipitation would occur, with a lowering in the pH; second, that the bleaching effect of the chlorine would remove the color.

Several difficulties were encountered in chlorinating the wastes in the laboratory. The quantity of wastes used was too small to compute the doses by weight of chlorine, and a stock solution of chlorinated water had to be depended upon.

A stable chlorine solution was prepared in a solution of sodium hydroxide by adjusting the pH to slightly above neutral. The accuracy of dosing by this method is doubtful, since it is entirely possible that all the chlorine is not liberated from the caustic solution when added to the alkaline wastes. Acidification of the wastes prior to chlorination would have obscured the results. This form of treatment will be investigated in the field where more reliable results can be obtained, since laboratory results have shown that chlorination of wastes is a possible form of treatment and should be considered further.

Standardization of the chlorine water used for dosing and determinations for chlorines were computed by titration with sodium thiosulphate in a potassium iodide solution.

Residual chlorine interfered with the oxygen-consumed determinations, and several attempts were made to eliminate this difficulty. Neutralization of the chlorine with sulphur dioxide gas, followed by the boiling of the sample to drive off the residual sulphur dioxide was found to be the only successful method of eliminating the error.

Sulphur Blue Dye Liquor

The sample was obtained from the East Durham Cotton Mill, Durham, N. C., and is the exhausted sulphur-blue dye liquor from the dyeing of raw cotton in a Chattanooga vacuum pan.

The characteristics of the waste were:

pH	10.2
Total Alkalinity	1,575 p.p.m.
Pheno. Alkalinity	350 p.p.m.
Color	62,500
Oxygen Consumed	1,656 p.p.m.

The results from chlorination of this waste are given in Table 13.

From the results obtained it is seen that a residual chlorine of about 60–80 p.p.m. must be obtained at the end of 30 minutes to produce satisfactory results. The correct dosage for the chlorine treatment lies between 22.2 pounds per thousand gallons and 44.4 pounds per thousand gallons, at a cost of between $0.341 and $0.682 per thousand gallons. There is no advantage to be gained from dosing higher than this range.

The most striking result obtained is the high color removal, a reduction of 99.8% being recorded for the recommended dose.

The sludge produced is of a very light color, being about the same shade as the supernatant liquor; i.e., only a tinge of yellow.

Direct Black Dye Liquor

The chlorination tests on this waste were qualitative only, being made by bubbling chlorine through the waste. The results are as follows:

TABLE 13

Treatment of Sulfur Blue Dye Liquor by Chlorination

Chlorine Doses		Chlorine Cost* Per 1000 Gal.	Results in p.p.m. After 30 Minutes Contact				
			Residual Chlorine	Oxygen Consumed		Color	
p.p.m.	Lbs./1000 Gal.			p.p.m.	% Red.	p.p.m.	% Red.
RAW	WASTE	—	—	1656.0	0.0	62,500	0.0
2,674.3	22.2	$0.341	46.55	472.5	71.5	3,750	94.0
5,348.6	44.4	0.682	68.20	470.0	71.5	100	99.8
6,016.5	49.9	0.773	68.20	378.4	77.2	85	99.8
6,685.6	55.5	0.860	88.50	378.0	77.2	58	99.85
8,022.9	66.5	1.030	115.20	360.0	78.3	50	99.9

* The cost data have been computed on the basis of tank car quantities. The market price of $1.55 per 100 pounds in tank car lots is of October 15, 1932.

	Oxygen Consumed	Color
Raw,	730	50,000
Chlorinated,	132	250
% Red.,	81.8	99.5

Upon chlorination a heavy precipitate was formed, bright orange in color, which settled rapidly. The supernatant liquor was clear and pale yellow in color.

Kier Liquor

Qualitative tests were also run on kier liquor, with the following results:

	pH	Oxygen Consumed	Color
Raw,	11.5	2,765	10,000
Chlorinated,	7.3	1,786	2,000
% Red.,	—	35.3	80.0

The kier liquor did not respond to chlorination as readily as the sulphur-blue and direct-dye liquors. The investigation will be continued, however, on a quantitative basis.

Conclusions:—Chlorination of waste kier liquor results in a better effluent than obtained by any other chemical treatment, but the results obtained up to the present time are not such that the effluent can yet be considered satisfactory.

D. Precipitation with Chlorinated Sludge

That it is possible to rejuvenate sludge by chlorination, and use the rejuvenated sludge as a coagulant, has been shown by preliminary experiments.

The chlorinated sludge, upon addition to the raw waste, produces a heavy floc which settles rapidly, leaving a clear supernatant liquor.

The sludge evidently serves the double purpose of holding the chlorine ion until the chlorinated sludge is added to the raw waste, where it is liberated, and at the same time providing nuclei for the formation of the floc.

No data is available on this work at present, but the investigation is being continued.

E. Recovery

Recovery of Indigo Dye from Wash Water

Since the wastes from the indigo dyeing process at the White Oak Mill, Greensboro, N. C., are large in quantity and are discharged continuously to waste during the operating day, they were chosen for our first attempt at recovery.

It was estimated by officials of the Proximity Mill that from 1 to 1½% of the dye used was lost in the wash waters. Since the amount of goods dyed is large (there being two machines with an average output at

the present time of 55,000 pounds per day) the possibility of recovering the dye from the wash water presented itself.

Although the recovered indigo, at the present market prices, may not pay for the cost of the process, due attention should be given to its value as partially paying for the treatment of the wastes.

The method of recovery was by reversing the process of preparing the dye bath. The waste was first oxidized by aeration, and then neutralized by sulfuric acid, the reduced indigo compound being soluble only in an alkaline solution.

The dye, thus thrown out of solution, finally settled out. The time required for settling was several hours, and the cost of acid was $0.172 per thousand gallons.

It was found, however, that, by recirculating precipitated dye and adding it to the raw waste along with the acid, a heavy floc resulted, which settled in 15 minutes. The cost of chemicals by this method was reduced to $0.086 per thousand gallons.

In order to pay for the chemicals, then, the amount of recovered dye would have to be 0.15 pounds per thousand gallons of waste treated.

Unfortunately, we have been unable as yet to determine the percentage of indigo in the sludge recovered. Impurities in the sludge interferred with the determination, and since the analysis was made only recently we have been unable to finish the analysis in time to submit a definite report on the results at this time.

IV. Conclusions

The following conclusions have been drawn from the results of the work conducted during the first six months of my appointment:

(1) The use of calcium chloride in doses of 16.68 pounds per thousand gallons, a mixing period of 15 minutes, and a retention period for sedimentation of one hour with continuous sludge removal, are recommended for the chemical treatment of indigo wash water.

(2) The treatment of sulfur jig wastes in combination with sewage by biological filtration is successful. A very satisfactory effluent has been obtained from 15% sulfur jig waste and 85% domestic sewage. A concentration of 15% waste is not the maximum that can be cared for by this method of disposal.

(3) Direct-black dye wastes, after biological filtration, responded to treatment with activated carbon for color removal. The effluent from the chemical precipitation of this waste does not react to activated carbon.

Biological filtration of direct-black dye liquor is not sufficient treatment in itself, the residual color still being too high.

(4) The results of biological filtration of kier liquor were very gratifying; a 10% concentration of waste with 90% sewage produced a good clear effluent. At the present time the filter is operating with a 20% concentration of kier liquor, but not sufficient data have been taken as yet for any conclusions to be drawn.

The results of the studies with biological filtration of the wastes has a direct bearing on the situation now existing in Greensboro, N. C. The city is suspending construction of a

$1,500,000 sewage-treatment plant which will serve the district in which the textile mills are located, because of the lack of information on methods of treating the combined wastes. The city is willing to assume the obligation of treating the wastes along with the domestic sewage if satisfactory methods of treatment can be developed. We hope that the results of our work will find application in the solution of the problem existing in Greensboro.

(5) Chlorination of sulfur-blue wastes was successful, an effluent practically free from color being obtained.

(6) Chlorination of direct-black dye liquor gave fair results. The per cent color reduction was 99.5, but the effluent was still not satisfactory because of the residual color.

(7) Kier liquor responds to chlorination much better than to chemical precipitation. The results obtained are encouraging, but not conclusive. The work is being continued. The effluent is, however, better than that obtained by any other treatment by chemicals which has come to our attention.[12, 10]

(8) It is possible to precipitate the raw wastes by the addition of chlorinated sludge.

V. Future Studies

(1) A complete study of the chemical precipitation of direct- and sulfur-dye waste liquors will be carried out as was done with indigo wash water.

(2) Methods of disposal of the sludge resulting from chemical precipitation are to be investigated. The recovery of the organic matter of the wastes by conversion to fuel or gas, and its use as a fertilizer will be incident to this work.

(3) The studies of disposal of the wastes by biological filtration are to be continued.

(4) The feasibility of the use of activated carbon filters as a final step for complete color removal is to be determined.

(5) The work on chlorination of the wastes is worthy of investigation, and will be continued.

(6) The use of chlorinated sludge vs. direct chlorination will be investigated.

(7) The work on the recovery of indigo dye is to be completed. Recovery of the waste liquors for conversion to fuel or heating gas, and the use of some of the dyes as fertilizers are to be attempted.

The work on the design of the field plant is being pushed as rapidly as possible, and construction will start when those plans are finished. Our efforts are now concentrated on getting the field plant into operation at the earliest date possible.

As a result of the first six months of laboratory study, we feel that we are ready to take the cited projects into the field for detailed study with the pilot plant.

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Abstracts and Bibliography

This section is compiled under the direction of the Committee on Abstracts and Bibliography: Prof. E. R. Schwartz, Chairman, Dr. W. E. Emley, Prof. Louis A. Olney and M. W. Weiss.

Abstracts

I. Fibres (Synthetic and Natural) and Fibre Analysis

Flax Fibre Structure and Quality. S. A. C. Caldwell. *Tex. Mfg.*, June, 1933, P. 217–218.

A review of work from a number of sources on the structure of the flax fibre as disclosed by the microscope. Few direct references to the literature are to be found, which is regrettable in view of the detailed work which has been done and which is here reviewed in general. One interesting figure showing the joining of cells and their structure is reproduced, together with the usual conventional cross-section drawing of the plant stem. (S)

Jute and Cotton Celluloses: Comparative Studies. J. K. Chowdbury and N. N. Basu. *J. Indian Chem. Soc.*, 1932, V. 9, P. 615–31.

Purified cellulose from jute and from cotton give practically identical results from a chemical point of view, that is, yield of cellulose acetate, glucose, cellulose octa-acetate and CH_3O– in methylated cellulose. There is a slight difference, however, in the behavior of the two celluloses when tested by means of alkalies of different concentrations with the subsequent formation of viscose. The difference is more prominent in the physical tests where the jute derivatives exhibit a higher surface tension, lower viscosity and greater solubility. Both jute and cotton celluloses consist of polyanhydro-glucose residues, each glucose unit being probably linked to another in exactly the same manner, but the number of glucose residues constituting a polyanhydro-glucose unit appears to be lower in the jute cellulose. (Copied complete from *Chem. Abs.*, 1933, V. 27, P. 3327.) (W)

Oxycellulose and Hydrocellulose: Determination. H. Somer and H. Markert. *Rev. gen. mat. color.*, 1931, V. 35, P. 273–4, 313–15; *Monatschr. Textil-Ind.*, 1931, V. 46, P. 132–3, 173–5; *Chem. Abs.*, 1933, V. 27, P. 3341.

Oxycellulose is formed from vegetable textile materials through the action of oxidizing agents, and hydrocellulose through that of acids, but

these often have similar reactions in the tests now in use. In the present work search is made for a test which is capable of detecting which of the two alteration products is present, and to what extent the change has taken place. Appearance (fluorescence) in ultra-violet light from a quartz mercury vapor lamp, change in viscosity of cuprammonium solution, the use of dyes, and various chemical methods, are compared and discussed. (W)

SILK: CHEMICAL STUDIES ON CHINESE. I. GLYCINE, ALANINE AND TYROSINE CONTENTS OF CHEKIANG SILK. P. P. T. Sak, Tsu-Sheng Ma and Shao-Yuan Ma. *J. Chinese Chem. Soc.*, 1933, V. 1, P. 17–22.

From 100 grams of Chekiang silk were prepared by the methods of Fischer and Abderhalden 35g. of glycine ester-hydrochloride, 16.5g. of alanine and 10.5g. of tyrosine. The amount of tyrosine is about the same as in Italian and Japanese silks, while the glycine and alanine contents are somewhat lower. (Copied complete from *Chem. Abs.*, 1933, V. 27, P. 3081.) (W)

WOOL KERATIN: STRUCTURE AND ITS RELATION TO SWELLING PHENOMENA. J. B. Speakman. *J. Intern. Soc. Leather Trades' Chem.*, 1933, V. 17, P. 229–45; *Chem. Abs.*, 1931, V. 25, P. 1387, 4460, 5773.

Swelling of wool in water and alcohols is predominantly intermicellar; molecules of the swelling agent must be small enough to penetrate intermicellar spaces. Swelling facilitates stretching because of separation of micelles, and decreased attraction between parallel molecules within the micelle due to the intramicellar swelling agent. Relative decrease in resistance to stretching is determined by molecular weight and dielectric constant of the swelling medium. Swelling in acids, as deduced from decrease in the resistance of the fibre to extension, increases with decreasing pH for all acids down to pH = about 1; at lower pH swelling increases with decreasing dissociation constant of the acid. This is explained in terms of the Procter-Wilson theory as due to depression of the dissociation of the acid within the fibre by a common anion from the protein-acid salt. The zone of minimum swelling of wool is pH = 5 to 7. Acid-alkali titration of wool is a back-titration of salt linkages between histidine, lysine and arginine with glutamic and aspartic acids. Acid swelling facilitates extension by rupture of such linkages by acid and by separation of peptide chains by osmotic pressure. Heat treatment changes salt linkages to peptide linkage. Increased water absorption above about 40° is attributed to opening up of some unidentified linkage, possibly one involving oxygen. (Copied complete from *Chem. Abs.*, 1933, V. 27, P. 3131.) (W)

II. YARNS AND FABRICS

RAYON FABRICS: TENSION CONDITIONS AND THEIR IMPORTANCE IN TREATMENT AND FINISHING. W. Weltzien and W. Coordt. *Seide*, 1932, V. 37, P. 276–83, 304–14, 348–61, 392–5.

Method and apparatus are described for obtaining an extensibility curve for rayon with increasing load. A difference is observed between the extensibility curve of a normal fibre and the same one which has been swollen without tension in water and dried without tension, although the tensile strength or break point may be the same. For fibres which have

abnormal strains the dry extensibility curve will show considerable variation but after the wetting-out treatment the curve is similar to a normal fibre and repeated tests group the curves closely. In a study of viscose rayon warp threads, those from defective fabrics (striated dyeing, etc.) show abnormal dry extensibility curves and the abnormality may arise from strains imparted during the warp sizing or directly from the cross-winding of the cone especially if the lap makes adjustment to atmospheric moisture conditions impossible or incomplete. Viscose filling threads show in general the same properties but acetate rayon warp threads rarely exhibit a change in extensibility under the same conditions. (Copied complete from *Chem. Abs.*, 1933, V. 27, P. 3081–2.) (W)

III. Chemical and Other Processing (Not Otherwise Classifiɛd)

Dye Mixtures: Use of Stannous Chloride in Evaluation of. J. A. Kime. *J. Ind. & Eng. Chem., Anal. Ed.*, 1933, V. 5, P. 151–2.

Stannous chloride and titanous chloride are desirable reducing agents in testing dyes. The former is selective in its action and the stannic ions formed do not react with the titanous ions in the titration of a second dye. Among the permitted food dyes, those of the triphenylmethane group are stable towards $SnCl_2$ and some azo dyes are reduced easily by $SnCl_2$. A simple mixture of one dye from each of these groups can, therefore, be analyzed by titrating first with $SnCl_2$ and later with $TiCl_3$. (Copied complete from *Chem. Abs.*, 1933, V. 27, P. 3080.) (W)

IV. Research Methods and Apparatus

Fibre Measurement: New Methods of. (Neue Wege der Fasermessung.) H. Meyer. *Mell. Textilber.*, Vol. 12, Nov., 1931, P. 683–686; Dec., 1931, P. 743–745.

The standardization tendencies at the International Wool Conference, Liege, 1930, are anticipated and the recent suggestions are given of Fraser-Roberts (determination of fineness of wool), Barker and King (combined micro-weighing and projection method), New and Alty (weighing method determining the weight of the elementary fibre), Krauter (weighing method) and attention is called to the 'Fibre Meter of the NWK' (Nordwolle Konzern) covered by the German patent 510,298. The instrument is designed for the measurement of the average fibre fineness as well as for the fibre length by optical (microscope) and weighing (torsion balance) devices. The features of the instruments are detailed, the performance of the different measurements and 6 formulae for the data are discussed at length. Limit of error amounts to only $\pm$ 4 figs. (Copied complete from *Instruments* Feb., 1933, P. A8.) (S)

Rove Testing: Jute, Flax, etc. Communicated by James F. Low and Co., Limited, Monifieth. *Tex. Mfr.*, May, 1933, P. 196–197.

Brief outlines of techniques employed for the determination for free moisture, measured after air drying at room temperature; fibre length, computed from the weight per unit length along a rove (a special cutter

is illustrated); and regularity, determined by the weights of known lengths, and the variation expressed as a per cent above or below the average. (S)

VISCOSE RAYON: SIGNIFICANCE OF TENSILE TESTS OF. J. M. Preston. *Tex. Mfr.*, June, 1933, P. 227–228.

Producers and users carry out tensile tests on rayon yarns, but the meaning of these tests is imperfectly understood. The relations of the strengths and extensions to the structure, degradation, humidity, and other factors is discussed in this article and compared with those of other yarns. It is recommended to test rayon yarns both wet and dry and after a mild alkaline scour. (S)

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